

**WEST**[Generate Collection](#)[Print](#)**Search Results - Record(s) 1 through 3 of 3 returned.****1. Document ID: JP 2000281864 A**

L3: Entry 1 of 3

File: DWPI

Oct 10, 2000

DERWENT-ACC-NO: 2001-074473

DERWENT-WEEK: 200117

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TITLE: Polyacetal resin composition comprising polyacetal resin and clay interlaminar compound(s) derived from amino compound(s) and expandable silicate(s), useful as molding material.

PRIORITY-DATA: 1999JP-0091681 (March 31, 1999)

## PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
JP 2000281864 A	October 10, 2000		012	C08L059/00

INT-CL (IPC): C08 K 3/34; C08 K 5/17; C08 K 9/04; C08 L 59/00; C09 C 1/40

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	PMC
Draw Desc	Image									

**2. Document ID: JP 04239049 A**

L3: Entry 2 of 3

File: DWPI

Aug 26, 1992

DERWENT-ACC-NO: 1992-335433

DERWENT-WEEK: 199241

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TITLE: Acetal! polymer compsn. with high heat ageing properties - in which acetal! homopolymer is mixed with hindered phenolic antioxidant, amino cpd. and formaldehyde scavenger

PRIORITY-DATA: 1991JP-0001400 (January 10, 1991)

## PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
JP 04239049 A	August 26, 1992		007	C08L059/00

INT-CL (IPC): C08K 5/13; C08K 5/17; C08L 59/02

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	PMC
Draw Desc	Image									

**3. Document ID: JP 04239048 A**

L3: Entry 3 of 3

File: DWPI

Aug 26, 1992

Substantially the same procedure as in Example 1. The results are shown in Table 1.

#### Comparative Example 3

Substantially the same procedure as in Example 1 was repeated except that calcium stearate D-2 was used instead of calcium stearate D-1. The results are shown in Table 1.

#### Comparative Example 4

Substantially the same procedure as in Example 1 was repeated except that polyethylene C-1 was not used. The results are shown in Table 1.

#### Comparative Examples 5 and 6

Substantially the same procedure as in Example 1 was repeated except that the amount of polyethylene C-1 was changed as indicated in Table 1. The results are shown in Table 1.

#### EXAMPLES 5 and 6

Substantially the same procedure as in Example 1 was repeated except that the amount of polyethylene C-1 was changed as indicated in Table 1. The results are shown in Table 1.

#### EXAMPLES 7 to 9

Substantially the same procedure as in Example 1 was repeated except that the polyethylenes indicated in Table 2 were used instead of polyethylene C-1. The results are shown in Table 2.

#### Comparative Examples 7 and 8

Substantially the same procedure as in Example 1 was repeated except that the polyethylenes indicated in Table 2 were used instead of polyethylene C-1. The results are shown in Table 2.

#### Comparative Example 9

Substantially the same procedure as in Example 8 was repeated except that calcium stearate D-2 was used instead of calcium stearate D-1. The results are shown in Table 2.

#### EXAMPLE 10

100 parts by weight of oxymethylene copolymer A-1, 0.3 part by weight of sterically hindered phenol B-1, 0.3 part by weight of polyethylene C-8 and 0.1 part by weight of calcium stearate D-1 were blended together in a Henschel mixer, and the resultant blend was melt-kneaded and extruded by means of a twin-screw extruder having an L/D ratio of 30 and a diameter of 40 mm, under conditions that the cylinder temperature was 210° C., the revolution rate of the screw was 100 rpm and the discharge rate was 30 kg/hour. The extruded molten strands were cooled and pelletized, thereby obtaining pellets. With respect to the thus obtained pellets, an extrusion molding test was carried out. The results are shown in Table 2.

#### EXAMPLES 11 and 12

Substantially the same procedure as in Example 1 was repeated except that the sterically hindered phenols indi-

calated in Table 2 were used instead of sterically hindered phenol B-1. The results are shown in Table 2.

#### EXAMPLE 13

100 parts by weight of oxymethylene copolymer A-1, 0.3 part by weight of sterically hindered phenol B-1, 0.3 part by weight of polyethylene C-3 and 0.1 part by weight of calcium laurate D-3 were blended together in a Henschel mixer, and the resultant blend was melt-kneaded by means of a twin-screw extruder having an L/D ratio of 30 and a diameter of 40 mm, under conditions that the cylinder temperature was 210° C., the revolution rate of the screw was 100 rpm and the discharge rate was 30 kg/hour. The extruded molten strands were cooled and pelletized, thereby obtaining pellets. With respect to the thus obtained pellets, an extrusion molding test was carried out. The results are shown in Table 2.

#### Comparative Example 10

Substantially the same procedure as in Example 13 was repeated except that calcium laurate D-4 was used instead of calcium laurate D-3. The results are shown in Table 2.

#### EXAMPLE 14

100 parts by weight of oxymethylene copolymer A-1, 0.3 part by weight of sterically hindered phenol B-1, 0.2 part by weight of polyethylene C-3, 0.05 part by weight of calcium stearate D-1 and 0.05 part by weight of melamine E-1 were blended together in a Henschel mixer, and the resultant blend was melt-kneaded and extruded by means of a twin-screw extruder having an L/D ratio of 30 and a diameter of 40 mm, under conditions that the cylinder temperature was 210° C., the revolution rate of the screw was 100 rpm and the discharge rate was 30 kg/hour. The extruded molten strands were cooled and pelletized, thereby obtaining pellets. With respect to the thus obtained pellets, an extrusion molding test was carried out. The results are shown in Table 3.

#### EXAMPLES 15 and 16

Substantially the same procedure as in Example 14 was repeated except that the formaldehyde-reactive substances indicated in Table 3 were used instead of melamine E-1. The results are shown in Table 3.

#### Comparative Example 11

100 parts by weight of oxymethylene copolymer A-1, 0.3 part by weight of sterically hindered phenol B-1, 0.2 part by weight of polyethylene C-1, 0.05 part by weight of calcium stearate D-1 and 0.5 part by weight of polyethylene glycol F-1 were blended together in a Henschel mixer, and the resultant blend was melt-kneaded and extruded by means of a twin-screw extruder having an L/D ratio of 30 and a diameter of 40 mm, under conditions that the cylinder temperature was 210° C., the revolution rate of the screw was 100 rpm and the discharge rate was 30 kg/hour. The extruded molten strands were cooled and pelletized, thereby obtaining pellets. With respect to the thus obtained pellets, an extrusion molding test was carried out. The results are shown in Table 3.

DERWENT-ACC-NO: 1992-335432

DERWENT-WEEK: 199241

COPYRIGHT 2002 DERWENT INFORMATION LTD

TITLE: Acetal! polymer compsn. with high heat ageing properties - in which acetal! polymer is mixed with hindered phenolic antioxidant and amino cpd,.

PRIORITY-DATA: 1991JP-0001399 (January 10, 1991)

## PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
JP 04239048 A	August 26, 1992		008	C08L059/00

INT-CL (IPC): C08K 5/13; C08K 5/17; C08L 59/00

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	PMC
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Term	Documents
POLYACETAL.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	14283
POLYACETALS.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	7647
AMINO.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	485710
AMINOES.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	1
AMINOS.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	478
AMINOE.DWPI,TDBD,EPAB,JPAB,USPT,PGPB.	111
COMPOUND[DWPI,TDBD,EPAB,JPAB,USPT,PGPB]	118262
CPD[DWPI,TDBD,EPAB,JPAB,USPT,PGPB]	136894
CPDS[DWPI,TDBD,EPAB,JPAB,USPT,PGPB]	79762
COMPOUNDS[DWPI,TDBD,EPAB,JPAB,USPT,PGPB]	80884
(POLYACETAL AND AMINO COMPOUND [TI]). USPT,PGPB,JPAB,EPAB,DWPI,TDBD.	3

[There are more results than shown above. Click here to view the entire set.](#)**Display Format:** -[Change Format](#)[Previous Page](#)[Next Page](#)

## Comparative Example 12

100 parts by weight of oxymethylene copolymer A-1, 0.3 part by weight of sterically hindered phenol B-1, 0.2 part by weight of polyethylene C-1, 0.05 part by weight of melamine E-1, 0.5 part by weight of polyethylene glycol F-1 and 0.1 part by weight of ethylene bisacryl amide F-2 were blended together in a Henschel mixer, and the resultant blend was melt-kneaded and extruded by means of a twin-screw extruder having an L/D ratio of 30 and a diameter of 40 mm, under conditions that the cylinder temperature was 210° C., the revolution rate of the screw was 100 rpm and the discharge rate was 30 kg/hour. The extruded molten strands were cooled and pelletized, thereby obtaining pellets. With respect to the thus obtained pellets, an extrusion molding test was carried out. The results are shown in Table 3.

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**Count**

**Set**  
**Name**  
result set

*DB=USPT,PGPB,JPAB,EPAB,DWPI,TDBD; PLUR=YES; OP=ADJ*

<u>L14</u>	l1 and l2 and reduced formaldehyde	7	<u>L14</u>
<u>L13</u>	l1 and l2	1719	<u>L13</u>
<u>L12</u>	l1 and l3	1	<u>L12</u>
<u>L11</u>	l1 and l8	1	<u>L11</u>
<u>L10</u>	l1 and l4	1	<u>L10</u>
<u>L9</u>	l1 and l7	1	<u>L9</u>
<u>L8</u>	polyacetal near3 reduced formaldehyde	5	<u>L8</u>
<u>L7</u>	polyacetal near2 reduced formaldehyde	2	<u>L7</u>
<u>L6</u>	polyacetal near1 reduced formaldehyde	0	<u>L6</u>
<u>L5</u>	polyacetal near reduced formaldehyde	0	<u>L5</u>
<u>L4</u>	polyacetal near3 reduced formaldehyde	5	<u>L4</u>
<u>L3</u>	polyacetal near5 reduced formaldehyde	7	<u>L3</u>
<u>L2</u>	polyacetal	20778	<u>L2</u>
<u>L1</u>	monoethanolamine or diethanolamine or 2 amino 2 ethyl propanediol or 2 amino 2 methyl propanol or tris(hydroxymethyl)aminomethane or ethyl p aminobenzoate or methyl anthranilate or butyl m aminobenzoate	38147	<u>L1</u>

END OF SEARCH HISTORY

# DETAILED DESCRIPTION OF THE INVENTION

According to the present invention, there is provided an oxyethylene polymer resin composition comprising:

(A) 100 parts by weight of an oxyethylene polymer, (B) 0.05 to 2 parts by weight of a sterically hindered phenol compound,

(C) 0.01 to 5 parts by weight of a low-density polyethylene having a melt index of from 0.2 to 100 g/10 minutes, (D) 0.01 to 1.0 part by weight of a calcium salt of a fatty acid having 10 to 36 carbon atoms, wherein the calcium salt of the fatty acid has a calcium ion content of not larger than 50 ppm by weight and a chlorine ion content of not larger than 100 ppm by weight, and

(E) 0 to 2.0 parts by weight of at least one formaldehyde- reactive substance selected from the group consisting of a non-polymeric compound (E<sub>1</sub>) containing a formaldehyde- reactive nitrogen atom and a polymer (E<sub>2</sub>) containing a formaldehyde- reactive nitrogen atom.

For easy understanding of the present invention, the essential features and various preferred embodiments of the present invention are enumerated below.

1. An oxyethylene polymer resin composition comprising:

(A) 100 parts by weight of an oxyethylene polymer, (B) 0.05 to 2 parts by weight of a sterically hindered phenol compound,

(C) 0.01 to 5 parts by weight of a low-density polyethylene having a melt index of from 0.2 to 100 g/10 minutes, (D) 0.01 to 1.0 part by weight of a calcium salt of a fatty acid having 10 to 36 carbon atoms, wherein the calcium salt of the fatty acid has a calcium ion content of not larger than 50 ppm by weight and a chlorine ion content of not larger than 100 ppm by weight, and

(E) 0 to 2.0 parts by weight of at least one formaldehyde- reactive substance selected from the group consisting of a non-polymeric compound (E<sub>1</sub>) containing a formaldehyde- reactive nitrogen atom and a polymer (E<sub>2</sub>) containing a formaldehyde- reactive nitrogen atom.

2. The resin composition according to item 1 above, wherein the oxyethylene polymer (A) is at least one polymer selected from the group consisting of an oxy-methylene copolymer, a branched oxyethylene copolymer and a block copolymer of an oxyethylene copolymer.

3. The resin composition according to item 1 or 2 above, wherein the oxyethylene polymer (A) has a melt index of from 0.1 to 150 g/10 minutes.

4. The resin composition according to any one of items 1 to 3 above, wherein the phenol compound (B) is at least one polymer selected from the group consisting of triethylene glycol-bis-(3-(3-1-butyl-5-methyl-4-hydroxyphenyl)-propionate), pentaerythritol-tris-(3-(3-1-butyl-5-methyl-4-hydroxyphenyl)-propionate), N,N'-5-di-1-butyl-4-hydroxyphenyl-propionate, N,N'-hexamethylene-bis(3,5-di-1-butyl-4-hydroxyphenyl)-propionate, 1,6-hexanediol-bis-(3-(3-1-butyl-4-hydroxyphenyl)-propionate), and 3,9-bis-(2-(3-(3-1-butyl-4-hydroxy-5-methylphenyl)-propionyl)-propionyl)-1,1,1-trimethylolpropane-tris(3,5-di-1-butyl-4-hydroxyphenyl)-propionate.

5. The resin composition according to any one of items 1 to 4 above, wherein the hindered phenol compound (B) is present in an amount of from 0.1 to 1.0 part by weight, relative to 100 parts by weight of component (A).

6. The resin composition according to any one of items 1 to 5 above, wherein the low-density polyethylene (C) is at least

# 6

one polyethylene selected from the group consisting of a high pressure low-density polyethylene and a linear low-density polyethylene.

7. The resin composition according to item 6 above, wherein the high pressure low-density polyethylene has a density of from 0.91 to 0.93 g/cm<sup>3</sup> and the linear low-density polyethylene has a density of from 0.88 to 0.93 g/cm<sup>3</sup>.

8. The resin composition according to any one of items 1 to 8 above, wherein the low-density polyethylene (C) has a melt index of from 0.2 to 100 g/10 minutes.

9. The resin composition according to any one of items 1 to 8 above, wherein the low-density polyethylene (C) is a high pressure low-density polyethylene.

10. The resin composition according to any one of items 1 to 10 above, wherein the calcium salt of fatty acid (D) is at least one calcium salt selected from the group consisting of calcium laurate, calcium stearate and acid calcium behenate.

11. The resin composition according to any one of items 1 to 10 above, wherein the calcium salt of fatty acid (D) is present in an amount of from 0.02 to 0.5 part by weight, relative to 100 parts by weight of component (A).

12. The resin composition according to any one of items 1 to 12 above, wherein the calcium salt of fatty acid (D) is present in an amount of from 0.02 to 0.5 part by weight, relative to 100 parts by weight of component (A).

13. The resin composition according to any one of items 1 to 12 above, wherein:

the non-polymeric compound (E<sub>1</sub>) containing a formaldehyde- reactive nitrogen atom is at least one compound selected from the group consisting of an amino-substituted triazine compound, a polyamide resin and an acrylamide polymer.

14. The resin composition according to item 13 above, wherein the amino-substituted triazine compound is a melamine, the amino-substituted triazine compound and the acrylamide polymer is at least one polymer selected from the group consisting of a poly-β-alanine and a polyacrylamide.

15. The resin composition according to any one of items 1 to 14 above, wherein the formaldehyde- reactive substance (E) is present in an amount of from 0 to 1.0 part by weight, relative to 100 parts by weight of component (A).

16. An extrusion molded article obtained from the resin composition of item 1 above, which is a rod or a sheet.

17. A shaped article obtained by cutting the extrusion molded article of item 16 above.

The present invention will now be described below in detail.

In the present invention, examples of oxyethylene polymers used as component (A) include:

(1) an oxyethylene homopolymer consisting essentially of oxyethylene units, which is obtained by polymerizing a formaldehyde monomer or a cyclic oligomer of formaldehyde, such as a formaldehyde trimer (trioxane) or a formaldehyde tetramer (tetraoxane);

(II) an oxyethylene copolymer containing 0.1 to 20 % by weight of oxyalkylene units having 2 to 8 carbon atoms, which is obtained by copolymerizing a formaldehyde monomer or a cyclic oligomer of formaldehyde, such as trioxane or tetraoxane, with a

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**Search Results - Record(s) 1 through 7 of 7 returned.****1. Document ID: US 20020019469 A1**

L14: Entry 1 of 7

File: PGPB

Feb 14, 2002

PGPUB-DOCUMENT-NUMBER: 20020019469  
PGPUB-FILING-TYPE: new  
DOCUMENT-IDENTIFIER: US 20020019469 A1

TITLE: Polyacetal resins with reduced formaldehyde odor

PUBLICATION-DATE: February 14, 2002

## INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY	RULE-47
Mori, Hiroshi	Tochigi-ken	DE	JP	
Kassal, Robert James	Wilmington		US	
Shinohara, Kenichi	Tochigi-ken		JP	

US-CL-CURRENT: 524/247

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	RWC
Draw Desc	Image										

**2. Document ID: US 4379862 A**

L14: Entry 2 of 7

File: USPT

US-PAT-NO: 4379862  
DOCUMENT-IDENTIFIER: US 4379862 A

TITLE: Process for the preparation of polyurethane resins using low molecular weight polyhydroxyl compounds prepared by the condensation of formaldehyde

DATE-ISSUED: April 12, 1983

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Wagner; Kuno	Leverkusen			DE

US-CL-CURRENT: 521/158; 521/170, 528/76, 528/80, 528/85

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	RWC
Draw Desc	Image										

**3. Document ID: US 4341909 A**

The concentration of the Irgadox 1076 (when used) was approximately 0.25%. UV-Check AM-340 was included in some blends as a synergist at a concentration of 0.22%.

The pellets were injection molded in a Newbury 25-ton injection molding machine at 400° F. into 7½"×½" tensile bars. A control sample containing only Irganox 1076 and control samples containing Irganox 1076 and Ciba-Geigy's Chimasorb 944 and Tinuvin 770 were included for comparison.

The test specimens were placed in a QUV accelerated weathering tester (Q Panel Company) for various exposure times. The QUV operated with an 8-hour light cycle using UV-B bulbs at 60° C. and a 4-hour condensation cycle at 50° C. Samples were placed in the QUV

The tensile bars were pulled on an instrumented Instron (Model 4204) according to ASTM Procedure 638. The minimum QUV exposure time required to obtain a brittle break in the Instron test was determined. A result

was considered a prime break when the tensile bar snapped before 15% elongation was obtained. The QLV time interval required to generate spotting and clouding of the surface of the tensile bars was also noted.

UV under the same conditions for 60 days. The tensile bars were then pulled on the Instron. A brittle break was considered a failure and greater than 15% elongation was considered passing. The results are summarized in Table 1.

The results are summarized in Table 1.

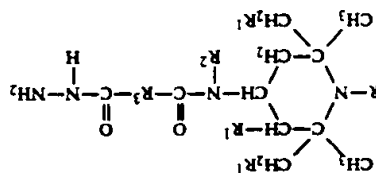
TABLE I

Stabilization of Polypropylene with HALS Hydrazides of Formula I									
HALS COM- POUND	Exam- ple # (Ea- st)	POLY- PROPYLENE TENSILE STRENGTH	IRGANOX 1076	UV-CHECK AM-50	DAYS TO SPOTTING	DAYS TO BRIGHTEN	DAYS TO BRITTLE	PASS- FAIL	80 DAYS IN QUVA
VIII	III	2.3	445	—	>50 <60	>50 <60	>50 <60	Pass	
IX	III	2.3	445	—	>50 <60	>50 <60	>50 <60	NT	
X	III	2.3	445	—	>70	>70	>70	Pass	
XI	III	2.3	445	1.1	>70	>70	>70	NT	
XII	I	2.55	445	—	35	>40 <50	>40 <50	Pass	
XIII	VII	2.7	445	—	>35 <40	>40 <50	>40 <50	Pass	
XIV	VII	2.7	445	1.1	>35 <40	>40 <50	>40 <50	NT	
XV	VII	2.7	445	—	>70	>70	>70	Pass	NT
CONTROLS									
XVI	A	—	445	1.1	6	5	5	Fail	
XVII	A	2.85	445	1.1	35	>15 <25	>15 <25	Fail	
XVIII	B	2.30	445	1.1	>35	>20 <25	>20 <25	Fail	

A = Camacho 94d Ciba-Geigy N,N'-Bis[2,2,6,6-tetramethyl-1-piperidinyl]-1,6-hexanediamine polymer with 2,4,6-trichloro-1,3,5-trimethyl-1,2-pentane.  
B = Timuvin 770 Ciba Geigy Di-(2,2,6,6-tetramethyl-1-piperidinyl) sebacate  
UV-Chex 1076 = Ciba-Geigy octadecyl 3,5-di-t-butyl-4-hydroxybenzoate  
NT = Not Tested

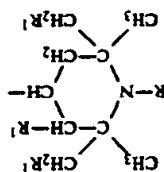
**We claim:**

1. A process for preparing a hydrazide of formula I



comprising reacting a half ester-half amide of formula

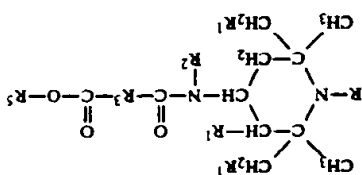
R<sub>3</sub> is a direct bond, an alkylene or branched alkylene diethylidene or 1 to 14 carbons, an alkylene diethylidene or 2 to 10 carbons, an oxydialkylene or thiodialkylene diethylidene or 4 to 10 carbons or a substituted or unsubstituted -o-, m- or p-phenylene diethylidene where the substituents may be lower alkyl, lower



with an equivalent amount or a slight excess of hydrazine or hydrazine hydrate at a sufficient temperature and for sufficient length of time to convert the half ester-half amide of formula IV to the hydrazide of formula I, where

R is hydrogen, oxy, hydroxy, alkyl of 1 to 20 carbons, alkenyl of 3 to 8 carbons, alkynyl of 3 to 8 carbons, aralkyl of 7 to 12 carbons, aliphatic acyl of 2 to 10 carbons, unsubstituted aryl acyl of 7 to 13 carbons, alkoxy, carbonyl of 2 to 9 carbons, arylalkoxy, carbonyl of 7 to 15 carbons, alkyl-, aryl-, cycloalkyl- or aralkyl-substituted carbamoyl of 2 to 13 carbons, 2-cyanoalkyl, hydroxyalkyl of 1 to 6 carbons, epoxy-alkyl of 3 to 10 carbons or a polyalkylene oxide group of 4 to 30 carbons;  
R<sup>1</sup> is hydrogen or lower alkyl of 1 to 4 carbons;  
R<sup>2</sup> is hydrogen, alkyl of 1 to 10 carbons, cycloalkyl of 5 to 12 carbons, aralkyl of 7 to 12 carbons, aryl of 6 to 12 carbons, 2-cyanoethyl or a radical of formula

४।३।३



A



L14: Entry 3 of 7

File: USPT

US-PAT-NO: 4341909

DOCUMENT-IDENTIFIER: US 4341909 A

TITLE: Preparation of low molecular weight polyhydroxyl compounds

DATE-ISSUED: July 27, 1982

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Schneider; Gottfried	Leverkusen			DE
Wagner; Kuno	Leverkusen			DE
Muller; Hanns P.	Leverkusen			DE

US-CL-CURRENT: 568/863; 521/158, 528/55

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
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## 4. Document ID: US 4301310 A

L14: Entry 4 of 7

File: USPT

US-PAT-NO: 4301310

DOCUMENT-IDENTIFIER: US 4301310 A

TITLE: Process for the preparation of low molecular weight polyhydroxyl compounds

DATE-ISSUED: November 17, 1981

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Wagner; Kuno	Leverkusen			DE

US-CL-CURRENT: 568/863; 568/388, 568/463

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	KWIC
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## 5. Document ID: US 4247653 A

L14: Entry 5 of 7

File: USPT

US-PAT-NO: 4247653

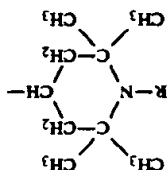
DOCUMENT-IDENTIFIER: US 4247653 A

TITLE: Process for the preparation of low molecular weight polyhydroxyl compounds

DATE-ISSUED: January 27, 1981

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Wagner; Kuno	Leverkusen			DE



10 R<sup>3</sup> is a direct bond or alkylene diradical of 1 to 14

8. The process of claim 7 wherein the initial step further comprises

(a) reacting the amine of formula II and the mono ester acid chloride of formula III in a solvent selected from the group consisting of inert hydrocarbon, chlorinated hydrocarbon and ether;

(b) separating the amine hydrochloride;

(c) isolating the intermediate half ester-half amide of formula IV; and

(d) dissolving the isolated intermediate half ester-half amide of formula IV in a polar solvent selected from the group consisting of methanol, ethanol, propanol, isopropanol and ethylene glycol.

9. The process of claim 8, wherein the amine of formula II is 4-amino-2,2,6,6-tetramethylpiperidine and the mono ester acid chloride of formula III is ethyl oxalyl chloride or ethyl succinyl chloride.

10. The process of claim 9, wherein the mono ester acid chloride of formula III is ethyl oxalyl chloride or ethyl succinyl chloride.

11. The process of claim 10 wherein the reaction between the intermediate half ester-half amide of formula IV and hydrazine hydrate is conducted in methanol at a temperature between 20° C. and 30° C.

12. The process of claim 1 further comprising an initial step of reacting an amine of formula II

with a dialkyl or diphenyl diester of formula VI

to form an intermediate half ester-half amide of formula VII

wherein

R<sup>1</sup> is hydrogen, methyl or acetyl,

R<sup>2</sup> is hydrogen, alkyl of 1 to 10 carbons, or a radical of formula

of formula

wherein

R<sup>3</sup> is a direct bond or alkylene diradical of 1 to 14

carbons,

R<sup>5</sup> is methyl, ethyl or phenyl.

8. The process of claim 7 wherein the initial step further comprises

(a) reacting the amine of formula II and the mono ester acid chloride of formula III in a solvent selected from the group consisting of inert hydrocarbon, chlorinated hydrocarbon and ether;

(b) separating the amine hydrochloride;

(c) isolating the intermediate half ester-half amide of formula IV; and

(d) dissolving the isolated intermediate half ester-half amide of formula IV in a polar solvent selected from the group consisting of methanol, ethanol, propanol, isopropanol and ethylene glycol.

9. The process of claim 8, wherein the amine of formula II is 4-amino-2,2,6,6-tetramethylpiperidine and the mono ester acid chloride of formula III is ethyl oxalyl chloride or ethyl succinyl chloride.

10. The process of claim 9, wherein the mono ester acid chloride of formula III is ethyl oxalyl chloride or ethyl succinyl chloride.

11. The process of claim 10 wherein the reaction between the intermediate half ester-half amide of formula IV and hydrazine hydrate is conducted in methanol at a temperature between 20° C. and 30° C.

12. The process of claim 1 further comprising an initial step of reacting an amine of formula II

with a dialkyl or diphenyl diester of formula VI

to form an intermediate half ester-half amide of formula VII

wherein

R<sup>1</sup> is hydrogen, methyl or acetyl,

R<sup>2</sup> is hydrogen, alkyl of 1 to 10 carbons, or a radical of formula

of formula

wherein

R<sup>3</sup> is a direct bond or alkylene diradical of 1 to 14

carbons,

R<sup>5</sup> is methyl, ethyl or phenyl.

alkoxy, hydroxy, bromo, chloro, mercapto or

lower alkylmercapto; and

R<sup>5</sup> is lower alkyl of 1 to 6 carbons or phenyl.

2. The process of claim 1 wherein

R is hydrogen, methyl or acetyl,

R<sup>1</sup> is hydrogen,

R<sup>2</sup> is hydrogen, alkyl of 1 to 10 carbons, or a radical of formula

of formula

wherein

R<sup>3</sup> is a direct bond or alkylene diradical of 1 to 14

carbons,

R<sup>5</sup> is methyl, ethyl or phenyl.

3. The process of claim 2, further comprising

(a) conducting the reaction in a polar solvent selected from the group consisting of methanol, ethanol, propanol, isopropanol and ethylene glycol; and

(b) isolating the compound of formula I by crystallization of the compound of formula I from the polar solvent or by evaporation of the polar solvent.

4. The process of claim 3 wherein R, R<sup>1</sup> and R<sup>2</sup> are hydrogen, R<sup>3</sup> is a direct bond and R<sup>5</sup> is methyl or ethyl.

5. The process of claim 4 wherein the polar solvent is methanol or ethanol, the temperature is between 10° C. and 30° C., and the hydrazide of formula I is isolated by crystallization from the polar solvent.

6. The process of claim 2, further comprising an initial step of reacting an excess of an amine of formula II

with a mono ester acid chloride of formula III

wherein

R is hydrogen or methyl,

with a mono ester acid chloride of formula III

wherein

R<sup>1</sup> is hydrogen or methyl,

R<sup>2</sup> is hydrogen, alkyl of 1 to 10 carbons, or a radical of formula

of formula

wherein

R<sup>3</sup> is a direct bond or alkylene diradical of 1 to 14

carbons,

R<sup>5</sup> is methyl, ethyl or phenyl.

3. The process of claim 2, further comprising

(a) conducting the reaction in a polar solvent selected from the group consisting of methanol, ethanol, propanol, isopropanol and ethylene glycol; and

(b) isolating the compound of formula I by crystallization of the compound of formula I from the polar solvent or by evaporation of the polar solvent.

4. The process of claim 3 wherein R, R<sup>1</sup> and R<sup>2</sup> are hydrogen, R<sup>3</sup> is a direct bond and R<sup>5</sup> is methyl or ethyl.

5. The process of claim 4 wherein the polar solvent is methanol or ethanol, the temperature is between 10° C. and 30° C., and the hydrazide of formula I is isolated by crystallization from the polar solvent.

6. The process of claim 2, further comprising an initial step of reacting an excess of an amine of formula II

with a mono ester acid chloride of formula III

wherein

R is hydrogen or methyl,

with a mono ester acid chloride of formula III

wherein

R<sup>1</sup> is hydrogen or methyl,

R<sup>2</sup> is hydrogen, alkyl of 1 to 10 carbons, or a radical of formula

of formula

wherein

R<sup>3</sup> is a direct bond or alkylene diradical of 1 to 14

carbons,

R<sup>5</sup> is methyl, ethyl or phenyl.

3. The process of claim 2, further comprising

(a) conducting the reaction in a polar solvent selected from the group consisting of methanol, ethanol, propanol, isopropanol and ethylene glycol; and

(b) isolating the compound of formula I by crystallization of the compound of formula I from the polar solvent or by evaporation of the polar solvent.

4. The process of claim 3 wherein R, R<sup>1</sup> and R<sup>2</sup> are hydrogen, R<sup>3</sup> is a direct bond and R<sup>5</sup> is methyl or ethyl.

5. The process of claim 4 wherein the polar solvent is methanol or ethanol, the temperature is between 10° C. and 30° C., and the hydrazide of formula I is isolated by crystallization from the polar solvent.

6. The process of claim 2, further comprising an initial step of reacting an excess of an amine of formula II

with a mono ester acid chloride of formula III

wherein

R is hydrogen or methyl,

with a mono ester acid chloride of formula III

wherein

R<sup>1</sup> is hydrogen or methyl,

R<sup>2</sup> is hydrogen, alkyl of 1 to 10 carbons, or a radical of formula

of formula

wherein

R<sup>3</sup> is a direct bond or alkylene diradical of 1 to 14

carbons,

R<sup>5</sup> is methyl, ethyl or phenyl.

US-CL-CURRENT: 521/158; 252/182.27, 252/71, 521/172, 521/173, 554/223, 554/227,  
560/115, 560/158, 560/26, 568/388, 568/463, 568/623, 568/678, 568/861, 568/863

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Draw Desc	Image	Draw Desc	Image
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6. Document ID: US 4219508 A

L14: Entry 6 of 7

File: USPT

US-PAT-NO: 4219508

DOCUMENT-IDENTIFIER: US 4219508 A

TITLE: Process for the preparation of low molecular weight polyhydroxyl compounds

DATE-ISSUED: August 26, 1980

INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Wagner; Kuno	Leverkusen			DE

US-CL-CURRENT: 568/387; 252/364, 252/73, 435/243, 435/247, 516/DIG.1, 516/DIG.2,  
521/107, 521/116, 521/158, 521/164, 521/168, 528/85, 549/417, 549/450, 549/453,  
549/488, 554/223, 560/198, 560/263, 562/553, 562/561, 564/473, 568/388, 568/463,  
568/496, 568/679, 568/723, 568/833, 568/852, 568/863

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Draw Desc	Image	Draw Desc	Image
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7. Document ID: KR 2001112405 A WO 200059993 A1 EP 1171519 A1 US  
20020019469 A1

L14: Entry 7 of 7

File: DWPI

Dec 20, 2001

DERWENT-ACC-NO: 2001-024449

DERWENT-WEEK: 200239

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TITLE: New polyacetal resin composition useful in molded articles comprises at least one odor reducing additive e.g. tris(hydroxymethyl)aminomethane or ethyl p-aminobenzoate

INVENTOR: KASSAL, R J; MORI, H ; SHINOHARA, K

PRIORITY-DATA: 1999US-0287432 (April 7, 1999), 2001US-0852383 (May 10, 2001)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
KR 2001112405 A	December 20, 2001		000	C08L059/00
WO 200059993 A1	October 12, 2000	E	018	C08K005/00
EP 1171519 A1	January 16, 2002	E	000	C08K005/00
US 20020019469 A1	February 14, 2002		000	C08L001/00

INT-CL (IPC): C08 K 5/00; C08 K 5:3445; C08 L 1/00; C08 L 59/00; C08 K 5/00; C08 K 5:17; C08 K 5:3445; C08 K 5:17; C08 K 5/00



US006147146A

United States Patent [19] Patent Number: 6,147,146 Date of Patent: Nov. 14, 2000

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2-166151 6/1990 Japan  
6-212054 8/1994 Japan  
7-324155 12/1995 Japan  
92/07033 4/1992 WIPO  
97/12937 10/1997 WIPO

Primary Examiner—Krellion Sanders  
Attorney, Agent, or Firm—Birch, Stewart, Kolasch & Birch, LLP

# ABSTRACT

[57] Disclosed are an oxymethylene polymer resin composition comprising an oxymethylene polymer, a sterically hindered phenol compound, a low-density polyethylene having a melt index of 0.2 to 100 g/10 minutes, a calcium salt of a fatty acid having 10 to 36 carbon atoms, wherein the calcium salt of a fatty acid has a calcium ion content of not larger than 50 ppm by weight and a chlorine ion content of not larger than 100 ppm by weight, and at least one formaldehyde- reactive substance selected from the group consisting of a non-polymeric compound containing a formaldehyde- reactive nitrogen atom and a polymer containing a formaldehyde- reactive nitrogen atom, and an extrusion molded article obtained from the above resin composition. The resin composition of the present invention is advantageously not only in that it has excellent extrusion moldability and exhibits excellent anti-slip properties when contacting the screw of an extruder during molding, but also in that an extrusion molded article produced therefrom has a remarkably reduced content of whitened portions and void portions comprising microvoids and hence has excellent mechanical properties and an excellent appearance.

# 17 Claims, No Drawings

## RESIN COMPOSITION

[75] Inventors: Mitsubishi Horio; Yoshiharu Seyama, both of Kurashiki, Japan  
[73] Assignee: Asahi Kasei Kogyo Kabushiki Kaisha, Osaka, Japan

[21] Appl. No. 09/269,263

[22] PCT Filed: Nov. 13, 1997

[86] PCT No. PCT/JP97/04141

[37] Date: Mar. 25, 1999

[102(c)] Date: Mar. 25, 1999

[87] PCT Pub. No. WO98/21280

[30] PCT Pub. Date: May 22, 1998

## Foreign Application Priority Data

Nov. 13, 1996 [JP] Japan 8-301782

[51] Int. Cl.<sup>7</sup> C08K 5/34; C08K 5/15;

C08K 5/20; C08K 5/10; C08K 5/04

[52] U.S. Cl. 524/100; 524/109; 524/223;

524/310; 524/400

[58] Field of Search 524/100, 109,

524/310, 400, 223

## References Cited

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5,519,075 5/1996 Matsumoto et al.

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**WEST****End of Result Set**

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L14: Entry 7 of 7

File: DWPI

Dec 20, 2001

DERWENT-ACC-NO: 2001-024449

DERWENT-WEEK: 200239

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TITLE: New polyacetal resin composition useful in molded articles comprises at least one odor reducing additive e.g. tris(hydroxymethyl)aminomethane or ethyl p-aminobenzoate

Basic Abstract Text (1):

NOVELTY - New polyacetal resin composition comprises at least one odor reducing additive. The additive is a low molecular weight primary or secondary amino compound of low volatility, succinimide, anthranilic acid and/or 4-amino benzoic acid.

Basic Abstract Text (2):

DETAILED DESCRIPTION - New polyacetal resin composition comprises a polyacetal resin (a) and at least one odor-reducing additive (b). (b) is a low molecular weight primary or secondary amino compound of low volatility, succinimide, anthranilic acid and/or 4-amino benzoic acid. The amino compound contains at least one amino group and at least two carbon atoms and has a weak basicity of Pkb 2-8. The resin composition has a formaldehyde concentration at room temperature of less than about 50% of the formaldehyde concentration of similar polyacetal compositions free of odor-reducing additive(s).

Basic Abstract Text (3):

An INDEPENDENT CLAIM is also included for reducing formaldehyde concentration from a molded part which involves blending (a) with (b) to form a blend with reduced formaldehyde concentration.

Basic Abstract Text (5):

ADVANTAGE - The composition has excellent heat stability, air aging and moldability as well as a reduced formaldehyde level for packaged resins and molded parts. The combination of the odor-reducing additive with a weak acidic imino compound provides a synergistic effect in further reducing the formaldehyde odor for packaged resins and molded parts.

Equivalent Abstract Text (1):

NOVELTY - New polyacetal resin composition comprises at least one odor reducing additive. The additive is a low molecular weight primary or secondary amino compound of low volatility, succinimide, anthranilic acid and/or 4-amino benzoic acid.

Equivalent Abstract Text (2):

DETAILED DESCRIPTION - New polyacetal resin composition comprises a polyacetal resin (a) and at least one odor-reducing additive (b). (b) is a low molecular weight primary or secondary amino compound of low volatility, succinimide, anthranilic acid and/or 4-amino benzoic acid. The amino compound contains at least one amino group and at least two carbon atoms and has a weak basicity of Pkb 2-8. The resin composition has a formaldehyde concentration at room temperature of less than about 50% of the formaldehyde concentration of similar polyacetal compositions free of odor-reducing additive(s).

Equivalent Abstract Text (3):

An INDEPENDENT CLAIM is also included for reducing formaldehyde concentration from a molded part which involves blending (a) with (b) to form a blend with reduced formaldehyde concentration.

(3,5-di-*t*-butyl-4-hydroxyphenyl)benzene, 3,9-bis[2-(3-(3-*t*-butyl-4-hydroxy-5-methylphenyl)-propionyloxy]-1,1-dimethyl-2,2'-undecane, 2,2'-methylene-bis[4-methyl-6-*t*-butylphenol], 1,1,3-tris(2-methyl-4-hydroxy-5-*t*-butylphenyl)butane, 1,6-hexanediol-bis[3-(3-*t*-butyl-4-hydroxyphenyl)-4-hydroxyphenyl]-propionate, *N,N'*-hexamethylene-bis[4-hydroxyphenyl]propionate, *N,N'*-hexamethylene-bis[3-*t*-butyl-4-hydroxy-5-methyl-2-hydroxyphenyl)-4-hydroxyphenyl]-propionate, 1,6-hexanediol-bis[3-(3,5-di-*t*-butyl-4-hydroxyhexamethylene-bis[3,5-di-*t*-butyl-4-hydroxy-5-methylphenyl)-4-hydroxyphenyl]-propionate], *N,N'*-hexamethylene-bis[3,5-di-*t*-butyl-4-hydroxy-5-methylphenyl)-4-hydroxyphenyl]-propionate and 3,9-bis[2-(3-(3-*t*-butyl-4-hydroxy-5-methylphenyl)-propionyloxy]-1,1-dimethyl-2,2'-undecane are preferred. These sterically hindered phenol compounds can be used individually or in combination.

The amount of the sterically hindered phenol compound used in the present invention is generally in the range of from 0.05 to 2.0 parts by weight, preferably from 0.1 to 1.0 part by weight, relative to 100 parts by weight of the oxyethylene polymer as component (A). When the amount of the sterically hindered phenol compound is smaller than 0.05 part by weight, satisfactory heat resistance and extrusion moldability cannot be obtained. When the amount of the sterically hindered phenol compound is larger than 2.0 parts by weight, the resin composition is likely to suffer marked discoloration in a molding machine during molding, so that the resultant molded article is not suitable for practical use.

As the low-density polyethylene component (C) of the resin composition of the present invention, use can be made of at least one polyethylene selected from the group consisting of a high pressure low-density polyethylene and a linear low-density polyethylene.

A high pressure low-density polyethylene is produced by radical polymerization under a high pressure, namely a pressure in the range of from 1,000 to 3,000 kg/cm<sup>2</sup>. During the radical polymerization for producing a high pressure low-density polyethylene, short branchings (such as ethyl branchings and butyl branchings) from a main chain occur by an intramolecular hydrogen abstraction reaction due to "back biting". Due to the presence of the short branchings, the obtained high pressure polyethylene has a low density. Also, during the radical polymerization, long branchings occur due to an intermolecular hydrogen abstraction reaction, so that the high pressure low-density polyethylene is caused to have long branchings having a length comparable to that of a main chain. The density of the high pressure low-density polyethylene is generally in the range of from 0.91 to 0.93 g/cm<sup>3</sup>.

A linear low-density polyethylene is produced by ionic polymerization. Specifically, a linear low-density polyethylene is produced by a method in which an ethylene monomer is copolymerized with an  $\alpha$ -olefin, such as 1-butene,

cyclic ether, such as ethylene oxide, propylene oxide, epichlorohydrin, 1,3-dioxolane or a diglycol formal; (III) a branched oxyethylene homopolymer obtained by polymerizing a formaldehyde monomer or a cyclic oligomer of formaldehyde, such as trioxane or tetraoxane, in the presence of a compound having, in a molecule thereof, a plurality of functional groups, such as hydroxyl groups, carboxyl groups, amino groups, acid anhydride groups, alkoxy groups and/or epoxy groups; (IV) a branched oxyethylene copolymer obtained by copolymerizing a formaldehyde monomer or a cyclic oligomer of formaldehyde, such as trioxane or tetraoxane, with a cyclic ether, such as ethylene oxide, propylene oxide, epichlorohydrin, glycol formal or diglycol formal, in the presence of a styrene type, an ester type, an amide type or a urethane type elastomer having a functional group (such as a hydroxyl group, a carboxyl group, an amino group, an acid anhydride group, an alkoxy group or an epoxy group) at one end thereof; and (V) a block copolymer of an oxyethylene homopolymer, obtained by polymerizing a formaldehyde monomer or a cyclic oligomer of formaldehyde, such as trioxane or tetraoxane, in the presence of a styrene type, an ester type, an amide type or a urethane type elastomer having a functional group (such as a hydroxyl group, a carboxyl group, an amino group, an acid anhydride group, an alkoxy group or an epoxy group) at one end thereof; and (VI) a block copolymer of an oxyethylene copolymer, obtained by copolymerizing a formaldehyde monomer or a cyclic oligomer of formaldehyde, such as trioxane or tetraoxane, with a cyclic ether, such as ethylene oxide, propylene oxide, epichlorohydrin, glycol formal or diglycol formal, in the presence of a styrene type, an ester type, an amide type or a urethane type elastomer having a functional group (such as a hydroxyl group, a carboxyl group, an amino group, an acid anhydride group, an alkoxy group or an epoxy group) at one end thereof or at both ends thereof.

Of these oxyethylene polymers, an oxyethylene copolymer of item (II) above, a branched oxyethylene copolymer of item (IV) above and a block copolymer of an oxyethylene copolymer of item (VI) above are preferred. An oxyethylene copolymer of item (II) above is more preferred.

The melt index (MI) of the oxyethylene polymer used in the present invention is generally from 0.1 to 150 g/10 minutes, preferably from 1 to 100 g/10 minutes. With respect to a copolymer type polyoxyethylene polymer, the commoner content thereof is generally from 0.1 to 20 mol, preferably from 0.3 to 10 mol, relative to 100 mol of the oxyethylene. In the present invention, the melt index is measured in accordance with ASTM-D1238-57T.

Examples of sterically hindered phenol compounds used as component (B) of the resin composition of the present invention include triethylene glycol-bis[3-(3-*t*-butyl-5-methyl-4-hydroxyphenyl)-propionate], 1,6-hexanediol-bis[3-(3,5-di-*t*-butyl-4-hydroxyphenyl)-propionate], [3-(3,5-di-*t*-butyl-4-hydroxyphenyl)-propionate], pentaerythritol-tetrakis[3-(3,5-di-*t*-butyl-4-hydroxyphenyl)-propionate], *N,N'*-hexamethylene-bis[3,5-di-*t*-butyl-4-hydroxy-5-methylphenyl)-4-hydroxyphenyl]-propionate, 3,5-di-*t*-butyl-4-hydroxybenzylphosphonate-diethyl ester, 1,3,5-trimethyl-2,4,6-tris

Equivalent Abstract Text (5):

ADVANTAGE - The composition has excellent heat stability, air aging and moldability as well as a reduced formaldehyde level for packaged resins and molded parts. The combination of the odor-reducing additive with a weak acidic imino compound provides a synergistic effect in further reducing the formaldehyde odor for packaged resins and molded parts.

Standard Title Terms (1):

NEW POLYACETAL RESIN COMPOSITION USEFUL MOULD ARTICLE COMPRISE ONE REDUCE ADDITIVE TRI  
HYDROXYMETHYL AMINOMETHANE ETHYL P

arachidonic acid, propionic acid and stearic acid and heptenic acid are

The calcium salt of a fatty acid having 10 to 36 carbon atoms, which is used as component (D) of the resin composition of the present invention, is insoluble in water and methanol, and therefore produces no calcium ion. However, a calcium salt of a fatty acid having 10 to 36 carbon atoms is generally produced according to the below-described production process, and such calcium salt of a fatty acid generally contains both calcium ions and chlorine ions. The calcium ions are derived from, for example,  $\text{Ca}(\text{OH})_2$  and the like, which are present as impurities, and the chlorine ions are derived from, for example,  $\text{NaCl}$ ,  $\text{CaCl}_2$  and the like, which are also present as impurities.

There is no particular limitation with respect to the method for producing the specific calcium salt of a fatty acid used in the present invention, as long as the obtained calcium salt of a fatty acid has a calcium ion content of substantially not larger than 50 ppm by weight and a chlorine ion content of substantially not larger than 100 ppm by weight. Specifically, for example, the calcium salt of a fatty acid used in the present invention can be obtained by a method in which a calcium salt of a fatty acid is produced by a reaction between a sodium salt of a fatty acid and calcium chloride. The obtained calcium salt of the fatty acid is repeatedly washed with water to remove calcium ions and chlorine ions until the calcium ion content and the chlorine ion content become 50 ppm by weight or less and 100 ppm by weight or less, respectively. When calcium hydroxide having a low fatty acid, the number of washing operations necessary for obtaining the calcium salt of a fatty acid can be decreased. The calcium ion content and the chlorine ion content of a calcium salt of a fatty acid can be determined by ion chromatography. For example, a calcium salt of a fatty acid is added to a methanol/water mixture (weight ratio of 1:1). Thereafter, the resultant is subjected to ion chromatography for determination of calcium ions and chlorine ions, if any. When the calcium ion content of component (D) is more than 50 ppm by weight and/or when the chlorine ion content of component (D) is more than 100 ppm by weight, problems arise not only in that the occurrence of whitened portions and void portions in an extrusion molded article is increased, but also in that discoloration tends to occur during the annealing of an extrusion molded article. Complete elucidation has not yet been made with respect to the reason why the occurrence of whitened portions and void portions in an extrusion molded article is increased when the calcium ion content requirement both defined in the present invention is not

1-hexene, 4-methylpentene-1 or 1-octene, which is employed in an amount of from several % to several tens % by weight, based on the weight of the ethylene monomer, thereby introducing short branchings into the main chain so as to decrease the density of the resultant polymer. The density of the linear low-density polyethylene is generally in the range of from 0.88 to 0.93 g/cm<sup>3</sup>.

Elucidation has not yet been made with respect to the mechanism in which the low-density polyethylene in the resin composition of the present invention serves to decrease the occurrence of whitened portions and void portions in an extrusion molded article obtained from the resin composition. However, it has been found that, when the low-density polyethylene in the resin composition of the present invention is replaced by a high-density polyethylene (which has substantially no short branches and substantially no long branches), the effect of the present invention cannot be achieved. From this result, it is presumed that the effect of the present invention has some relationship to the branching structure of the low-density polyethylene in the resin composition. On the other hand, it has also been found that, as the low-density polyethylene component, a high pressure low-density polyethylene is more effective for achieving the effect of the present invention than a linear low-density polyethylene. Therefore, it is also believed that the presence of long branchings in the low-density polyethylene is advantageous for achieving the effect of the present invention.

It is preferred that the low-density polyethylene has a melt index of from 0.2 to 100 g/10 minutes. It is more preferred that the low-density polyethylene has a melt index of from 0.3 to 50 g/10 minutes. When the melt index of the low-density polyethylene is smaller than 0.2 g/10 minutes, the suppression of the occurrence of whitened portions and void portions in an extrusion molded article is likely to become unsatisfactory. When the melt index of the low-density polyethylene is larger than 100 g/10 minutes, the occurrence of void portions is increased and the resin composition tends to slip on the screw of an extruder during molding. The amount of the low-density polyethylene is in the range of from 0.01 to 5 parts by weight, preferably from 0.05 to 3 parts by weight, relative to 100 parts by weight of the oxymethylene polymer as component (A). When the amount of the low-density polyethylene is less than 0.01 part by weight, the suppression of the occurrence of whitened portions and void portions in an extrusion molded article is likely to become unsatisfactory. When the amount of the low-density polyethylene is more than 5 parts by weight, unfavorable exfoliation and internal fine cracking tend to occur in an extrusion molded article.

The calcium salt of a fatty acid, used as component (D) of the resin composition of the present invention, is a calcium salt of a saturated or unsaturated fatty acid having 10 to 36 carbon atoms. The fatty acid may be substituted with a hydroxyl group. Examples of saturated fatty acids include capric acid, lauric acid, myristic acid, palmitic acid, stearic acid, arachidic acid, heptenic acid, lignocenic acid, cerotic acid, montanic acid, melissic acid and ceroplastic acid. Examples of unsaturated fatty acids include undecylenic acid, oleyic acid, elaidic acid, cetoleic acid, erucic acid, brassidic acid, sorbic acid, linoleic acid, linolenic acid, 1-hexene, 4-methylpentene-1 or 1-octene, which is employed in an amount of from several % to several tens % by weight, based on the weight of the ethylene monomer, thereby introducing short branchings into the main chain so as to decrease the density of the resultant polymer. The density of the linear low-density polyethylene is generally in the range of from 0.88 to 0.93 g/cm<sup>3</sup>.



**WEST**[Generate Collection](#)[Print](#)**Search Results - Record(s) 1 through 7 of 7 returned.****1. Document ID: US 20020019469 A1**

L14: Entry 1 of 7

File: PGPB

Feb 14, 2002

PGPUB-DOCUMENT-NUMBER: 20020019469  
PGPUB-FILING-TYPE: new  
DOCUMENT-IDENTIFIER: US 20020019469 A1

TITLE: Polyacetal resins with reduced formaldehyde odor

PUBLICATION-DATE: February 14, 2002

## INVENTOR-INFORMATION:

NAME	CITY	STATE	COUNTRY	RULE-47
Mori, Hiroshi	Tochigi-ken	DE	JP	
Kassal, Robert James	Wilmington		US	
Shinohara, Kenichi	Tochigi-ken		JP	

US-CL-CURRENT: 524/247

<a href="#">Full</a>	<a href="#">Title</a>	<a href="#">Citation</a>	<a href="#">Front</a>	<a href="#">Review</a>	<a href="#">Classification</a>	<a href="#">Date</a>	<a href="#">Reference</a>	<a href="#">Sequences</a>	<a href="#">Attachments</a>
<a href="#">Draw Desc</a>	<a href="#">Image</a>								

[RWD](#)**2. Document ID: US 4379862 A**

L14: Entry 2 of 7

File: USPT

US-PAT-NO: 4379862  
DOCUMENT-IDENTIFIER: US 4379862 A

TITLE: Process for the preparation of polyurethane resins using low molecular weight polyhydroxyl compounds prepared by the condensation of formaldehyde

DATE-ISSUED: April 12, 1983

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Wagner; Kuno	Leverkusen			DE

US-CL-CURRENT: 521/158, 521/170, 528/76, 528/80, 528/85

<a href="#">Full</a>	<a href="#">Title</a>	<a href="#">Citation</a>	<a href="#">Front</a>	<a href="#">Review</a>	<a href="#">Classification</a>	<a href="#">Date</a>	<a href="#">Reference</a>	<a href="#">Sequences</a>	<a href="#">Attachments</a>
<a href="#">Draw Desc</a>	<a href="#">Image</a>								

[RWD](#)**3. Document ID: US 4341909 A**

son with respect to the number of spherulites (having a diameter of 100  $\mu$ m or more) is made between an extrusion molded article produced from an oxymethylene polymer resin composition which satisfies both the above-mentioned calcium ion content and chlorine ion content requirements and an extrusion molded article produced from an oxymethylene polymer resin composition which does not satisfy any of the above-mentioned calcium ion content and chlorine ion content requirements). From this finding, it is presumed that the number of crystal nuclei in the resin composition influences the solidification speed of the resin composition during molding and, in turn, the solidification speed affects the occurrence of a whitened portion and a void portion in the resultant molded article.

In the present invention, it is preferred that the calcium ion content and chlorine ion content of the calcium salt (D) of a fatty acid are as low as possible, most advantageously 0 ppm by weight.

The amount of the calcium salt (D) of a fatty acid is generally in the range of from 0.01 to 1.0 part by weight, preferably from 0.02 to 0.5 part by weight, relative to 100 parts by weight of the oxymethylene polymer.

When the amount of the calcium salt (D) of a fatty acid is smaller than 0.01 part by weight, the effect of component (D) for improving the heat resistance of the resin composition is unsatisfactory. When the amount of the calcium salt (D) of a fatty acid is larger than 1.0 part by weight, vigorous discoloration occurs during the annealing of an extrusion molded article.

As component (E) of the resin composition of the present invention, use is made of at least one formaldehyde-reactive substance selected from the group consisting of a non-polymeric compound (E<sub>1</sub>) containing a formaldehyde-reactive nitrogen atom and a polymer (E<sub>2</sub>) containing a formaldehyde-reactive nitrogen atom. The non-polymeric compound (E<sub>1</sub>) is a monomeric compound or an oligomeric compound. Examples of non-polymeric compounds (E<sub>1</sub>) include (1) dicyandiamide and (2) amino-substituted triazine compounds. Examples of amino-substituted triazine compounds (2) include guanamine (2,4-diamino-sym-triazine), melamine (2,4,6-triamino-sym-triazine), N-butylmelamine, N-phenylmelamine, N,N-diphenylmelamine, N,N-diallylmelamine, N,N'-dimethylolmelamine, N,N',N''-trimethylolmelamine, benzoguanamine (2,4-diamino-6-phenyl-sym-triazine), 2,4-diamino-6-methyl-sym-triazine, 2,4-diamino-6-butyl-sym-triazine, 2,4-diamino-6-benzoyloxy-sym-triazine, 2,4-diamino-6-cyclohexyl-sym-triazine, 2,4-diamino-6-chloro-sym-triazine, 2,4-dioxo-6-amino-sym-triazine, and diamino-sym-triazine and N'-triacetamidoethylbenzoguanamine. Of these non-polymeric compounds (E<sub>1</sub>), dicyandiamide and melamine are preferred.

On the other hand, examples of polymers (E<sub>2</sub>) containing a formaldehyde-reactive nitrogen atom, which are usable as component (E) in the present invention, include (1) an amino-substituted triazine compound formaldehyde polymer, (2) a polyamide resin, and (3) an acrylamide polymer. Examples of amino-substituted triazine formaldehyde polymers include a melamine formaldehyde polymer and the like. Examples of polyamide resins (2) include nylon 4-6, nylon 6, nylon 6-6, nylon 6-10, nylon 6-12, nylon 6-6-6, nylon 6-6-6-10, and nylon 6-6-12. Examples of acrylamide polymers (3) include a homopolymer obtained by polymerizing acrylamide or a derivative thereof in the presence of a metal alcoholate, a homopolymer obtained by polymerizing acrylamide or a derivative thereof in the presence of a metal alcoholate, a copolymer obtained by polymerizing acrylamide or a derivative thereof with another vinyl monomer in the presence of a metal alcoholate, a copolymer obtained by polymerizing acrylamide or a derivative thereof with another vinyl monomer in the presence of a radical polymerization catalyst, a copolymer obtained by polymerizing acrylamide or a derivative thereof with another vinyl monomer in the presence of a radical polymerization catalyst and a copolymer obtained by polymerizing acrylamide or a derivative thereof with another vinyl monomer in the presence of a radical polymerization catalyst can be produced in accordance with the methods disclosed in Examined Japanese Patent Application Nos. 6-12259 corresponding to U.S. Pat. No. 5,015,707, 5-87096 and 5-47568 and Unexamined Japanese Patent Application Laid-Open Specification No. 3-234729. A homopolymer obtained by polymerizing acrylamide or a derivative thereof in the presence of a radical polymerization catalyst and a copolymer obtained by polymerizing acrylamide or a derivative thereof with another vinyl monomer in the presence of a radical polymerization catalyst can be produced in accordance with the method disclosed in Unexamined Japanese Patent Application Laid-Open Specification No. 3-28260.

The weight average molecular weight of the polymer (E<sub>2</sub>) containing a formaldehyde-reactive nitrogen atom is preferably 500 or more, more preferably 5,000 or more.

The formaldehyde-reactive substance (E) of the resin composition of the present invention exhibits an effect of improving the heat resistance of the resin composition when the molding temperature is higher than usual or when the processing speed is very low.

The amount of the formaldehyde-reactive substance (E) in the resin composition of the present invention is 0 to 2.0 parts by weight, preferably 0 to 1.0 part by weight, relative to 100 parts by weight of the oxymethylene polymer as component (A). When the amount of the formaldehyde-reactive substance (E) is more than 2.0 parts by weight, the resin composition suffers discoloration during the residence of the composition in a molding machine.

In the present invention, various additives used for conventional oxymethylene polymers can be used. Examples of additives include a weathering stabilizer (for example, a light stabilizer), a lubricant (mold release agent), a pigment and the like. These additives can be used individually or in combination. These additives can be used in an amount usually employed in the art.

As light stabilizers, (I) a benzotriazole type compound, (II) an oxalic anilide type compound and (III) a hindered amine type compound are preferred.

Examples of benzotriazole type compounds (I) include 2-(2-hydroxy-5'-methyl-phenyl)benzotriazole, 2-(2-

L14: Entry 3 of 7

File: USPT

US-PAT-NO: 4341909

DOCUMENT-IDENTIFIER: US 4341909 A

TITLE: Preparation of low molecular weight polyhydroxyl compounds

DATE-ISSUED: July 27, 1982

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Schneider; Gottfried	Leverkusen			DE
Wagner; Kuno	Leverkusen			DE
Muller; Hanns P.	Leverkusen			DE

US-CL-CURRENT: 568/863; 521/158, 528/55

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	RMIC
Draw Desc	Image									

## 4. Document ID: US 4301310 A

L14: Entry 4 of 7

File: USPT

US-PAT-NO: 4301310

DOCUMENT-IDENTIFIER: US 4301310 A

TITLE: Process for the preparation of low molecular weight polyhydroxyl compounds

DATE-ISSUED: November 17, 1981

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Wagner; Kuno	Leverkusen			DE

US-CL-CURRENT: 568/863; 568/388, 568/463

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	RMIC
Draw Desc	Image									

## 5. Document ID: US 4247653 A

L14: Entry 5 of 7

File: USPT

US-PAT-NO: 4247653

DOCUMENT-IDENTIFIER: US 4247653 A

TITLE: Process for the preparation of low molecular weight polyhydroxyl compounds

DATE-ISSUED: January 27, 1981

## INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Wagner; Kuno	Leverkusen			DE

[illegible]

US-CL-CURRENT: 521/158; 252/182.27, 252/71, 521/172, 521/173, 554/223, 554/227,  
560/115, 560/158, 560/26, 568/388, 568/463, 568/623, 568/678, 568/861, 568/863

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	PMO
Draw Desc	Image									

## 6. Document ID: US 4219508 A

L14: Entry 6 of 7

File: USPT

US-PAT-NO: 4219508

DOCUMENT-IDENTIFIER: US 4219508 A

TITLE: Process for the preparation of low molecular weight polyhydroxyl compounds

DATE-ISSUED: August 26, 1980

### INVENTOR-INFORMATION:

NAME	CITY	STATE	ZIP CODE	COUNTRY
Wagner; Kuno	Leverkusen			DE

US-CL-CURRENT: 568/387; 252/364, 252/73, 435/243, 435/247, 516/DIG.1, 516/DIG.2,  
521/107, 521/116, 521/158, 521/164, 521/168, 528/85, 549/417, 549/450, 549/453,  
549/488, 554/223, 560/198, 560/263, 562/553, 562/561, 564/473, 568/388, 568/463,  
568/496, 568/679, 568/723, 568/833, 568/852, 568/863

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	PMO
Draw Desc	Image									

## 7. Document ID: KR 2001112405 A WO 200059993 A1 EP 1171519 A1 US 20020019469 A1

L14: Entry 7 of 7

File: DWPI

Dec 20, 2001

DERWENT-ACC-NO: 2001-024449

DERWENT-WEEK: 200239

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TITLE: New polyacetal resin composition useful in molded articles comprises at least one odor reducing additive e.g. tris(hydroxymethyl)aminomethane or ethyl p-aminobenzoate

INVENTOR: KASSAL, R J; MORI, H ; SHINOHARA, K

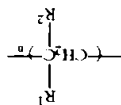
PRIORITY-DATA: 1999US-0287432 (April 7, 1999), 2001US-0852383 (May 10, 2001)

### PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
KR 2001112405 A	December 20, 2001		000	C08L059/00
WO 200059993 A1	October 12, 2000	E	018	C08K005/00
EP 1171519 A1	January 16, 2002	E	000	C08K005/00
US 20020019469 A1	February 14, 2002		000	C08L001/00

INT-CL (IPC): C08 K 5/00; C08 K 5:3445; C08 L 1/00; C08 L 59/00; C08 K 5/00; C08 K 5:17; C08 K 5:3445; C08 K 5:17; C08 K 5/00

The olefin compound having an average degree of polymerization of from 10 to 500, which is usable as a lubricant (mold release agent), is represented by the following formula (1):



(1)

wherein each of  $R_1$  and  $R_2$  independently represents a hydrogen atom, an alkyl group, an aryl group or an alkoxy group, and  $n$  represents an average degree of polymerization and is from 10 to 500.

Examples of alkyl groups include an ethyl group, a propyl group, a butyl group, a hexyl group, an octyl group, a decyl group, a lauryl group, a cetyl group and a stearyl group. Examples of aryl groups include a phenyl group, a p-butylphenyl group, a p-octylphenyl group, a p-nonylphenyl group, a benzyl group, a p-butylbenzyl group, a tolyl group and a xylol group. Examples of ether groups include an ethoxy group, a propoxy group and a butyloxy group. Examples of olefin monomers, such as

ethylene, propylene, 1-butene, 2-butene, isobutylene, 1-pentene, 2-pentene, 2-methyl-1-butene, 3-methyl-1-butene, 2-methyl-2-butene, 1-hexene, 2,3-dimethyl-2-butene, 1-heptene, 1-octene, 1-nonene and 1-decene, and diolefin monomers, such as allene, 1,2-butadiene, 1,3-butadiene, 1,3-pentadiene, 1,4-pentadiene, 1,5-hexadiene and cyclopentadiene. Alternatively, the olefin compound may be a copolymer obtained by copolymerizing 2 or more types of monomers selected from these olefin and diolefin monomers. When the olefin compound is obtained by polymerizing a diolefin monomer, from the viewpoint of increasing the heat stability of the olefin compound, it is preferred to use a modified olefin compound which is obtained by subjecting the olefin compound to a conventional hydrogenation treatment so as to decrease the number of carbon-carbon unsaturated bonds in the olefin compound to a number as small as possible.

It is necessary that the average degree of polymerization (n) of olefin monomer units constituting the olefin compound be in the range of from 10 to 500, preferably from 15 to 300. When the average degree (n) of polymerization is less than 10, the long-term lubrication properties of the olefin compound tend to become low and mold deposit problems tend to occur. When the average degree of polymerization (n) is more than 500, the initial lubrication properties of the olefin compound tend to become low.

As preferred examples of silicone oils usable as lubricants (mold release agents), there can be mentioned polydimethylsiloxane and polymethyl phenyl siloxane, both represented by the following formula (2):

release agents), examples of alcohols used for forming an ester with a dicarboxylic acid include saturated and unsaturated alcohols, such as methyl alcohol, ethyl alcohol, propyl alcohol, n-butyl alcohol, isobutyl alcohol, t-butyl alcohol, n-amyl alcohol, 2-pentanol, n-heptyl alcohol, n-octyl alcohol, n-nonyl alcohol, lauryl alcohol, myristyl alcohol, cetyl alcohol, stearyl alcohol and benzyl alcohol. Examples of dicarboxylic acids used for forming an ester with an alcohol include a monoester and a diester of oxalic acid, malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, undecanoic acid, brassidic acid, maleic acid, fumaric acid and glutaric acid.

As fatty acid amides usable as lubricants (mold release agents), there may be mentioned fatty acid amides obtained by reacting fatty acids having 16 or more carbon atoms with aliphatic amines or aliphatic diamines. Examples of fatty acids used for forming a fatty acid amide include palmitic acid, isopalmitic acid, stearic acid, isostearic acid, behenic acid, lignoceric acid, cerotic acid, heptacosanoic acid, monolanic acid, melissic acid, lauric acid, cetoleic acid and erucic acid. Examples of amines and diamines used for forming fatty acid amides include ammonia and ethylene diamine. Examples of fatty acid amides include stearyl amide, palmityl amide, oleyl amide, methylheptadecylamide, ethylheptadecylamide and ethylheptadecylamide.

Polyoxyalkylene glycols usable as lubricants (mold release agents) are classified into classes (I) to (III). The polyoxyalkylene glycols of class (I) are polycondensates of class (I) include polyethylene glycol, polypropylene glycol, and a block polymer of polyethylene glycol and polypropylene glycol. The preferred degree of polymerization of these polyoxyalkylene glycols is in the range of from 5 to 1,000, more advantageously from 10 to 500. The polyoxyalkylene glycols of class (II) are ethers obtained from a polyoxyalkylene glycol of class (I) and an aliphatic alcohol. Examples of polyoxyalkylene glycols of class (II) include polyethylene glycol oleyl ether (degree of polymerization of ethylene oxide: 5 to 50), polyethylene glycol cetyl ether (degree of polymerization of ethylene oxide: 5 to 20), polyethylene glycol stearyl ether (degree of polymerization of ethylene oxide: 5 to 30), polyethylene glycol lauryl ether (degree of polymerization of ethylene oxide: 5 to 30), polyethylene glycol indocyl ether (degree of polymerization of ethylene oxide: 5 to 30), polyethylene glycol nonyl ether (degree of polymerization of ethylene oxide: 2 to 100) and polyethylene glycol octyl phenyl ether (degree of polymerization of ethylene oxide: 4 to 50). The polyoxyalkylene glycols of class (III) are esters obtained from a polyoxyalkylene glycol of class (I) and a higher fatty acid. Examples of polyoxyalkylene glycols of class (III) include polyethylene glycol monolaurate (degree of polymerization of ethylene oxide: 2 to 30), polyethylene glycol monostearate (degree of polymerization of ethylene oxide: 2 to 50) and polyethylene glycol monooleate (degree of polymerization of ethylene oxide: 2 to 10).